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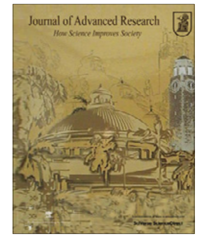

ORIGINAL ARTICLE

Effect of endodontic irrigation and dressing procedures on the shear bond strength of composite to coronal dentin

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$Ca(OH)_2$

Abstract This study aimed to evaluate the effects of three sodium hypochlorite (NaOCl)-endodontic irrigation procedures used alone or in combinations with two intermediate dressing materials on bond strengths of two adhesive composite systems to coronal dentin. Surfaces were treated with NaOCl or NaOCl–Glyde-File-Prep (H_2O_2 and EDTA) with or without chlorhexidine (CHX) as a final rinse. Intermediate dressing materials of calcium hydroxide ($Ca(OH)_2$) and sodium perborate (SP) were combined with surface treatments. Surface treatment groups (n = 10/group) included (1) distilled water (control), (2) 5.25% NaOCl (30 min), (3) NaOCl/Glyde (30 min), (4) NaOCl/Glyde (30 min) + CHX (2 min), (5) NaOCl/Glyde (30 min) + $Ca(OH)_2$ (5 days) + CHX (2 min), and (6) NaOCl/Glyde (30 min) + SP (9 days) + CHX (2 min). For each surface treatment group, dentin shear bond strengths of two different composite systems (Excite/Tetric Flow Chroma, [EX/TFC], and Clearfil Protect Bond/Protect Liner F [PB/PLF]) were evaluated. Median shear bond strengths (EX/TFC, PB/PLF) for each surface treatment group in MPa were (1) 21, 18; (2) 26, 18; (3) 21, 17; (4) 22, 16; (5) 17, 11; and (6) 14, 11, respectively. NaOCl significantly increased the bond strength of EX/TFC (p < 0.05), but did not significantly affect that of PB/PLF. The use of NaOCl/Glyde with CHX did not significantly affect EX/TFC (p > 0.05), whereas it significantly decreased PB/PLF (p < 0.05). $Ca(OH)_2$ and SP significantly decreased the bond strengths of both adhesive systems (p < 0.05). Adhesion to coronal dentin is dependent upon the irrigation regimen and the type of adhesive.

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Introduction

One of the primary goals of root canal treatment is to eliminate bacteria from the root canal system, which have been shown to be the etiology for apical periodontitis and endodontic failure. Furthermore, restoring of the tooth after root canal treatment should be capable of preventing recontamination of the root canal system [1]. The use of dentin adhesive systems and flowable composite resin has been advocated for use within the pulp chamber/access cavity in an attempt to work as an orifice barrier against microleakage [2]. However, endodontic materials inevitably contact the dentin of the access cavity during chemomechanical root canal preparation, therefore the dentin available for the coronal restoration may be modified and bonding procedures may also be affected [3–8].

Sodium hypochlorite (NaOCL) (5.25%) has been highly recommended as root canal irrigant due to its antibacterial and organic tissue dissolution properties [9]. However, it has been reported that it induces oxidations of some components in the dentin matrix [4,5] and also causes dentin subsurface deproteinization [10]. In literature, NaOCl irrigant was found to decrease the bond strengths of some adhesive systems [3–6] that was recovered by using reducing agents as sodium ascorbate [4,5], whereas it did not affect [3,5,7] or even increased [8] the bond strengths of other adhesives to coronal dentin.

The strategy of alternating irrigation with NaOCl and Ethylenediaminetetraacetic acid (EDTA) was found to maximize the effectiveness of the antisepsis, due to effective removal of the smear layer and increased dentin permeability [11,12]. Glyde File Prep (Dentsply De Trey GmbH, Germany) that contains hydrogen peroxide [H_2O_2] and EDTA was recommended to be used with NaOCl in alternating sequence [12]. It is claimed to facilitate endodontic shaping and cleansing due to the chelating EDTA and the effervescence through H_2O_2. NaOCl/Glyde irrigation procedure may also affect the bond strength to coronal dentin. Beside NaOCl and EDTA effects [13], residual H_2O_2 may negatively affect the bond strength [14]. It has been well reported that sodium perborate (SP); a H_2O_2-releasing agent used intra-coronally for tooth bleaching, reduces the bond strength of resin to coronal dentin [14].

The use of chlorhexidine (CHX) as a final rinse was found to enhance the disinfection of the root canal system [9,15]. In addition, encouraging recent studies [16,17] have demonstrated that the application of CHX to dentin decelerated the degradation of the hybrid layer exposed collagen fibrils. CHX was found not to produce detrimental effects to the immediate bond strength [16,17]. However, higher concentrations decreased the bond strength of some adhesive systems [18].

Calcium hydroxide ($Ca(OH)_2$) intracanal dressing was found to increase the healing rate in cases of periradicular lesions [19] and to promote apexification [20]. However, it may be slightly overextended into the pulp chamber during its application with some remnants in contact with coronal dentin. It has been reported that $Ca(OH)_2$ ions diffuse into the smear layer-free dentinal tubules [21]. It was found to decrease the bond strength to root dentin [22].

The purpose of this study was to evaluate the effects of three NaOCl-endodontic irrigation procedures used alone or in combinations with two intermediate dressing materials on shear bond strength of two adhesive composite systems to coronal dentin. NaOCl or NaOCl–Glyde-File-Prep (H_2O_2 and EDTA) surface treatments with or without CHX as a final rinse were evaluated. Intermediate dressing materials of $Ca(OH)_2$ and SP were also combined with surface treatments for evaluation.

Material and methods

One-hundred and twenty non-carious human third molars were collected in 0.5% Chloramine solution, cleaned and stored in distilled water (4 °C) for a maximum of 6 months [23]. The roots were removed, pulp tissues were removed and pulp chambers were washed with distilled water and filled with moist cotton pellets in order to avoid drying out of the specimens. Thus, dentin could simulate that of endodontically treated teeth (without intra-pulpal pressure). Then, the crowns were embedded in chemically cured acrylic resin. They were ground flat with a series of SiC-papers ending with 600 grit used on a polisher (Polimet, Buehler) to obtain a 4 mm diameter flat buccal dentin surfaces at 1.5–2.0 mm distance from the pulp (Fig. 1) [24].

Glass rings of 1.5 cm diameter were fixed onto the specimens using flowable composite to create a pulp chamber-like space (Fig. 1). Very thin strings of flowable composite were applied on the outer silane-treated surfaces of the borders of the glass rings. Thus, this ensured that the flowable composite would not contaminate the test area. The chambers could be closed by acrylic covers, which were cemented to the borders of the glass rings using temporary cement (Temp bond NE, Kerr Italia S.p.A., Italy). These closed chambers were intended to keep $Ca(OH)_2$ and SP pastes in place for defined periods and to confine definite amounts of the irrigants to dentin.

The dentin specimens were randomly and equally divided into two main groups ($n = 60$) and bonded with two adhesive systems/corresponding flowable composite after irrigation procedures: (1) an etch and rinse adhesive system (Excite/Tetric Flow Chroma, [EX/TFC], Vivadent, Liechtenstein) and (2) a self-etching primer system with antibacterial properties (Clearfil Protect Bond/Protect Liner F [PB/PLF], Kuraray, Japan) (Table 1). Each group was further subdivided according to the tested endodontic procedures, into 6 groups of 10 specimens each (Fig. 2):

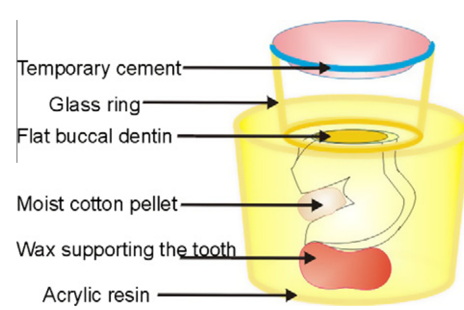

Fig. 1 A diagram showing the model developed to simulate the dentin of pulpless teeth and also the closed pulp chamber. The pulp tissue was excavated and replaced by moist cotton pellet. A glass ring was fixed onto the tooth specimen using flowable composite. The formed chamber was closed by an acrylic cover, which was cemented to the border of the glass ring using temporary cement.

Table 1 Materials information for bonding procedures.

Adhesive procedures	Excite/Tetric Flow Chroma (*EX/TFC*), Vivadent	Clearfil Protect bond/ Protect Liner F (*PB/PLF*), Kuraray
Etching	*Total Etch*™: (G07438)[a] 37% H_3PO_4[b]	
Priming	*Excite, Primer-adhesive*: (G02162) HEMA, DMA, phosphoric acid acrylate, silica, ethanol, initiators	*Primer*: (00002A) MDP, HEMA, hydrophilic dimethacrylate, MDPB, flouride, dl-Camphorquinone, N,N-Diethanol-p-toluidine, water)
Bonding		*Bond*: (00004A) MDP, Bis-GMA, HEMA, hydrophobic dimethacrylate, dl-Camphorquinone, N,N-Diethanol-p-toluidine, silanated colloidal silica
Flowable composite	*Tetric Flow Chroma*: (F53645) Bis-GMA, urethane dimethacrylate and TGDMA (35.0 wt.%). Inorganic fillers (64.6 wt.%). Catalysts, stabilizers and pigments (0.4 wt.%)	*Clearfil Liner Bond2 Protect Liner F*: (0056) Bis-GMA, TEGDMA, Silanated colloidal silica, prepolymerized organic filler, fluoride methyl methacrylate, camphorquinone

[a] Batch numbers are in parentheses.
[b] Chemical compositions were supplied from the manufacturers' manuals of products.

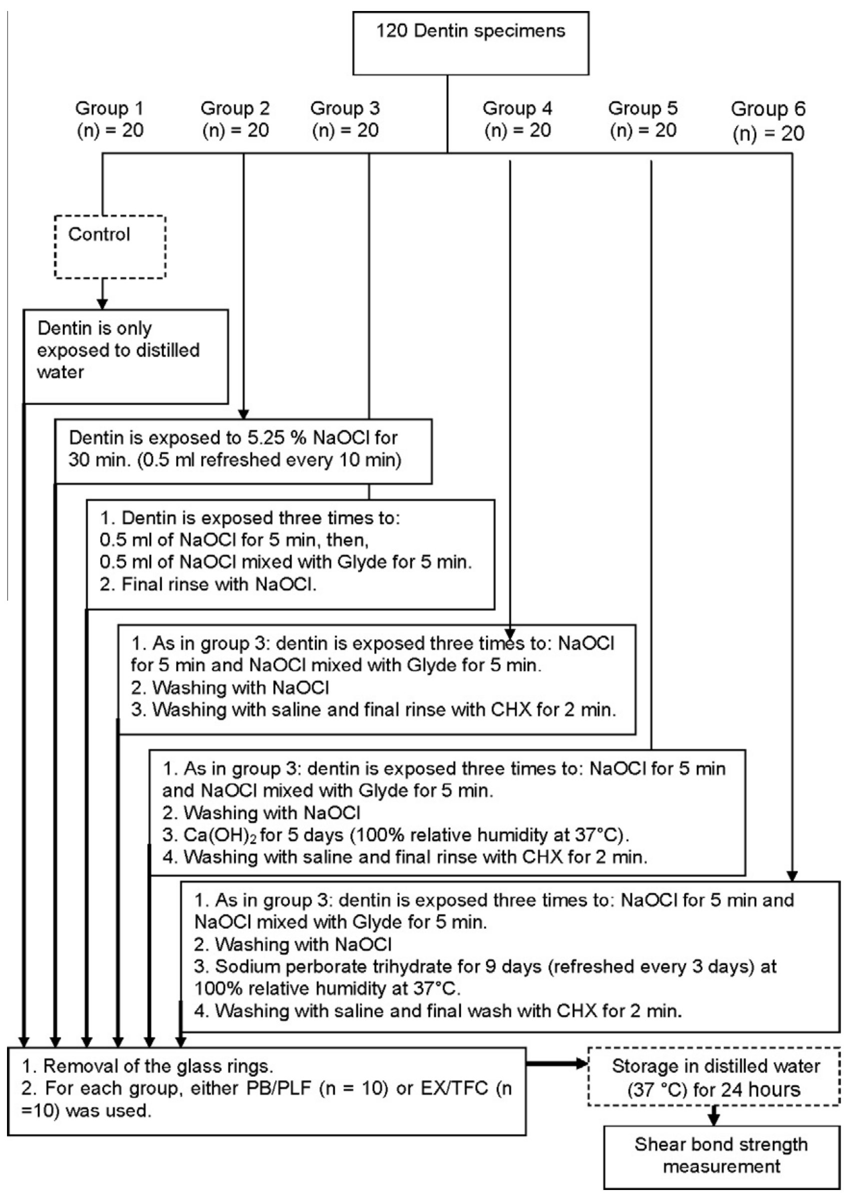

Fig. 2 Flow chart of the experimental methods. Glyde = Glyde File Prep.

Group 1: Dentin was exposed to distilled water as a control.
Group 2: Dentin was exposed to 5.25% NaOCl for 30 min (0.5 ml refreshed every 10 min; simulating the amount clinically in contact with coronal dentin).
Group 3: Dentin was exposed three times to: 0.5 ml NaOCl for 5 min, then, 0.5 ml NaOCl mixed with Glyde for 5 min in order to simulate chemo-mechanical root canal instrumentation with NaOCl/Glyde. Then, final rinse with NaOCl.
Group 4: In addition to chemo-mechanical debridement in group 3 (NaOCl/Glyde), the dentin was washed with saline and 0.5 ml CHX was applied as a final rinse (2 min). Saline was used to prevent any interaction between CHX and NaOCl [25].
Group5: In addition to chemo-mechanical debridement (NaOCl/Glyde), $Ca(OH)_2$ was applied for 5 days, removed by excavator and consecutively, dentin was washed with saline, then CHX as a final rinse.
Group 6: In addition to chemo-mechanical debridement (NaOCl/Glyde), SP paste (mixed with distilled water at 2 g/ml) was applied for 9 days (refreshed every three days), removed and the dentin was washed with saline and then CHX. The compositions of the endodontic materials used are listed in Table 2.

After dentin treatment with the aforementioned protocols, the glass rings were removed and the adhesive systems were applied in standardized circular dentin surfaces of 3 mm in diameter using flat metal rings with 3 mm inner diameter holes and 250 μm height. This flat metal ring assured accurately circular adhesive area without any flashes outside it. Using the Excite system, the dentin surface was acid etched for 15 s, rinsed for 30 s and dried with lint-free absorbent tissue. Then, the primer-adhesive was immediately applied on the dentin circular area with good agitation for 30 s and then gently air dried for 3 s. The primer-adhesive application was repeated in the same manner to finally obtain a glossy continuous layer and then light cured for 20 s. For the Clearfil Protect Bond system, the primer was applied on the dentin for 20 s and then air-thinned with gentle air stream. Then, the adhesive was applied on the dentin circular area with agitation for 20 s. Gentle air stream was applied to obtain a uniform adhesive layer which was light cured for 20 s. Flowable composite rods were built in two increments that were each light cured for 20 s, using a metal ring with a 3 mm diameter hole and 3 mm height. After storage of the specimens in distilled water at 37 °C for 24 h, shear bond strength was determined using a Material Testing Machine (Zwick 010, Ulm, Germany) at a cross head speed of 0.75 mm/min [26]. A chisel-shaped rod was placed at a distance of 70 μm from and parallel to the adhesive interface [27].

Medians and 25–75% quartiles were determined from ten specimens of each experimental group, and pairwise comparisons between groups were performed using the Mann–Whitney-Wilcoxon rank sum test (PC+, version 15, SPSS, Chicago, III., USA) at the 0.05 level of significance (α). The Error Rates Method was applied to detect significant differences between endodontic procedures and between adhesive systems in general by adjusting α to $\alpha^* = 1 - (1 - \alpha)^{1/K}$ (K = number of performed pairwise tests).

Results

The medians and the corresponding 25–75% quartiles of the shear bond strength values (MPa) for the groups are shown in Table 3. Results of pairwise comparisons between groups within each composite system and between each other are also shown in Table 3. The application of the Error Rates Method indicated a statistically significant influence of endodontic chemical debridement protocols and adhesive systems in general and irrespective of all other parameters.

As shown in Table 3, in comparison to the control group (distilled water) of EX/TFC, NaOCl significantly increased the bond strength of EX/TFC ($p = 0.009$), whereas NaOCl/Glyde and (NaOCl/Glyde)/CHX had no effect. The bond strength to NaOCl/Glyde-treated dentin was significantly lower than of NaOCl-treated dentin ($p = 0.009$). On the other hand, when compared to the control group, NaOCl and NaOCl/Glyde had no effect on the bond strength of PB/PLF while (NaOCl/Glyde)/CHX significantly decreased this bond strength ($p = 0.029$). (NaOCl/Glyde)/$Ca(OH)_2$/CHX significantly decreased the bond strength of EX/TFC ($p = 0.002$) and PB/PLF ($p = 0.000$). (NaOCl/Glyde)/SP/CHX significantly decreased the bond strength of EX/TFC ($p = 0.000$) and PB/PLF ($p = 0.000$).

Discussion

In the experimental design of the present study, the clinically applied irrigation and dressing procedures were simulated in groups, rather than separate endodontic materials that would obtain more clinically significant results. Studies investigated the effect of endodontic irrigants are sufficient [4–7,18], whereas those investigated endodontic irrigants in combinations are scarce [3,8]. The NaOCl/Glyde irrigation protocol

Table 2 Materials information for endodontic procedures.

Endodontic material	Chemical composition[a]
Hypochlorite SPEIKO® [SPEIKO®-Dr. Speier GmbH, Germany][b]	5.25% NaOCl
Glyde™ File Prep [Dentsply De Trey GmbH, Konstanz, Germany]	Ethylenediametetracetic acid (EDTA) and carbamide peroxide in water soluble base
Chlorhexidine [Sigma Chemicals Co., St. Louis, MO, USA]	0.2% Chlorhexidine digluconate
Hypocal® SN [Merz Dental GmbH, Germany]	100 g paste contain: 45 g Calcium hydroxide and 5 g Barium sulfate
Sodium perborate Bleaching agent [Merck, Darmstadt, Germany]	Sodium perborate trihydrate

[a] Chemical compositions were supplied from the manufacturers' manuals of products.
[b] Manufacturers of materials are in brackets.

Table 3 Dentin shear bond strengths for different endodontic procedures.

Experimental groups (n = 10)	Median bond strength values (25–75% quartiles) of *EX/TFC*	Median bond strength values (25–75% quartiles) of *PB/PLF*
(1) Distilled water	21 (20, 24)$^{A\ *,a\#}$	18 (17, 18)^{B,a}
(2) NaOCl	26 (24, 26)^{A,b}	18 (16, 19)^{B,ab}
(3) NaOCl/Glyde	21 (18, 23)^{A,a}	17 (13, 19)^{BD,ab}
(4) NaOCl/Glyde + CHX	22 (20, 27)^{A,ab}	16 (14, 17)^{BD,b}
(5) NaOCl/Glyde + $Ca(OH)_2$ + CHX	17 (15, 18)^{B,c}	11 (9, 14)^{D,c}
(6) NaOCl/Glyde + Na perborate + CHX	14 (13, 15)^{D,d}	11 (11, 13)^{C,c}

* Significant differences between the two composite systems are indicated by different capital letters.
\# Significant differences between groups within each composite system are indicated by different small letters.

may be optimal especially in cases of necrotic pulp with infected dentinal tubules [11]. The addition of CHX as a final rinse may be more prognostic in these cases [15].The use of NaOCl-alone irrigation protocol may be satisfactory in simple cases of vital pulp with irreversible pulpitis [28].

For testing the bond strength, buccal dentin surfaces were used to standardize the substrate as much as possible and as required by ISO-standard No. 11405 (2003) [23]. This reduces variation of data and enables studying the effect of endodontic irrigation protocols. Shear bond strength test was meticulously performed. 0.75 mm/min cross head speed was used as recommended in literature [26]. Due to the non-uniform stress distribution at the interface, the offset distance (distance from the dentin surface to the point of load application) was minimized to be 70 μm. This minimizes stress concentration inside the dentin substrate leading to less cohesive failure in dentin [27]. In the present study; partial cohesive failure in dentin did not exceed 30% of the specimens per group and the coefficient of variation ranged from 10% to 26% in all groups. In contrast, in the microtensile bond strength (μTBS) test (non-trimming technique), the stress distribution across the adhesive interface is uniform, however the tensile forces are applied on the adhesive interface passing through the two adherents resulting in some cohesive failure either in composite or in dentin [29]. In addition, producing mini-sticks requires cutting using slow speed saw that may either slightly decrease the bond strength or lead to premature failure of originally low bond strength specimens [30].

Previously, it was found that 5% NaOCl application to dentin for 60 min caused 80–100 μm dentin subsurface deproteinization [10]. It appeared as pores that were originally occupied by collagen fibers [31]. In the NaOCl groups of the present study, 5.25% NaOCl was applied on dentin for 30 min (refreshed every 10 min). The subsequent demineralization by the phosphoric acid of Excite and the primer of Protect Bond that was found to be 12 μm [32] and 0.5 μm [33], respectively, may result in a dentin subsurface deproteinized layer. It may be infiltrated by resin to a certain depth forming what is called a reverse hybrid layer [31]. This exposed collagen-free hybrid layer might positively affect the bond strength [31,34]. Besides, the previously reported NaOCl negative oxidizing effect on the bond strength [4,5] was found not to essentially affect all the adhesive systems [5].

The results of the present study showed that NaOCl-alone irrigation procedure significantly increased the bond strength using EX/TFC, whereas it did not significantly affect the bond strength using PB/PLF. In the literature, the μTBS of Excite to dentin was not affected by 10 min of 5% NaOCl [5], which indicated that NaOCl has little or no negative oxidizing effect on Excite. Therefore, in the present study, the longer NaOCl application period with increased deproteinization depth, may explain the increased bond strength. On the other hand, it was found in literature that the μTBS of Clearfil Protect Bond to dentin was significantly reduced by 30 s of 6% NaOCl; however, the use of reducing agents (5 or 10 s of accel or rosmarinic acid) successfully recovered it [4]. In the present study, the longer NaOCl application period may also explain the stable bond strength of PB/PLF. It should be mentioned that short NaOCl application periods [4,5] do not simulate the clinical irrigation period. Another study that used 1% NaOCl (refreshed every 5 min) for long period (1 h), increased the μTBS of a self etching adhesive system (XENO III, Dentsply) to dentin [8].

In NaOCl/Glyde groups of the present study, NaOCl and NaOCl–Glyde (EDTA + H_2O_2) were alternately applied on dentin three times (each for 5 min). It has been reported that the combined use of NaOCl and EDTA leads to interaction between them causing NaOCl to lose its tissue-dissolving capacity, whereas EDTA retained its calcium-chelating ability during the first 7 min of the reaction [13]. Therefore, in the present study, the alternating deproteinizing NaOCl and decalcifying EDTA may erode the coronal dentin surface [35] resulting, after application of the adhesive systems, in an almost normal hybrid layer. This may explain the results of the present study that showed that the bond strength of EX/TFC in NaOCl/Glyde group was significantly lower than NaOCl group but was in the range of the control group. The retention of H_2O_2 to dentin might be too low to affect the bond strength. On the other hand, the bond strength of PB/PLF in NaOCl/Glyde group was not significantly lower than the NaOCl- or the distilled water groups. However, the relatively high coefficient of variation (20%) of NaOCl/Glyde group, and the significant decrease of the bond strength of (NaOCl/Glyde)/CHX group in comparison to the distilled water group, may indicate the possible NaOCl and/or H_2O_2 negative effects on the bond strength of PB/PLF.

To date, no study has evaluated the effect of NaOCl/Glyde irrigation protocol on the bond strength to dentin. In the literature, 5.25% NaOCl for 5 min (refreshed every 1 min) followed by EDTA for 1 min resulted in a significant decrease of the μTBS of Clearfil SE Bond, Kuraray (self-etching primer system), whereas it did not affect that of XP-Bond, Dentsply (etch and rinse system) [3].

In the present study, no significant difference was found between (NaOCl/Glyde)/CHX and NaOCl/Glyde groups. Thus, the addition of CHX to NaOCl/Glyde irrigation procedure did not affect the bond strength of each adhesive system and this is in line with the literatures [16,17]. The significant decrease of the PB/PLF bond strength in (NaOCl/Glyde)/CHX group in comparison to distilled water group does not necessarily due to the contribution of 0.2% CHX. It has been reported that CHX may reduce the μTBS of some self-etch adhesives, due to a possible interaction between them. However, this was true at 2% concentration and not for 0.12% concentration [18].

The results of the present study showed that addition of $Ca(OH)_2$ for 5 days to the (NaOCl/Glyde)/CHX group significantly decreased the bond strength using either EX/TFC or PB/PLF. This is in line with previous findings [22,23]. Partial obliteration of the dentinal tubules by $Ca(OH)_2$ might act against proper etching and infiltration of adhesive resin into the dentinal tubules. In the present study, $Ca(OH)_2$ dressing decreased the bond strength of EX/TFC by 23%, which was less than the decrease observed with PB/PLF (31%). This may be explained by the phosphoric acid of EX/TFC that could decalcify larger thickness of the dentin surface when compared to the effect of the self-etching primer of PB/PLF, removing the highest concentration of the $Ca(OH)_2$ powder diffused inside the dentinal tubules. It may be suggested that if $Ca(OH)_2$ was inadvertently left in the coronal part, it should be carefully excavated. EDTA solution, which was recommended be used to eliminate the $Ca(OH)_2$ dressing from the root canal, may also be used during its excavation from the coronal part. Also, delaying of the immediate restoration with a wet cotton pellet inside the access cavity under a temporary filling might allow $Ca(OH)_2$ to diffuse out of the dentinal tubules.

The negative oxidizing effect of SP was confirmed in the present study. The addition of SP to (NaOCl/Glyde)/CHX group significantly decreased the bond strengths of both the adhesive systems and this in line with literature [14]. This may be explained by residuals of the oxygen forced inside the dentinal tubules during the long intracoronal bleaching period (9 days). Oxygen residuals may inhibit resin polymerization and also interfere with the resin infiltration [14]. Delaying of the restoration of the bleached tooth for two weeks was found to recover this reduced bond strength [14].

Conclusions

Based on the results of the present study, EX/TFC may be used within the pulp chamber after NaOCl-alone, NaOCl–Glyde-File-Prep or NaOCl–Glyde-File-Prep–CHX irrigation regimens tested in the present study. For PB/PLF, further investigations are needed to test if the application of a reducing agent after the irrigation procedure would be sufficient to obtain optimal bond strength. $Ca(OH)_2$ intermediate dressing should not be overextended into the pulp chamber. New methods for optimal removal of $Ca(OH)_2$ should be studied in order to enhance the bond strength after $Ca(OH)_2$ use.

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